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Palm Oil Mill Efluent - Zeolite Mixture Improves Acid and Base Cations in Acid Sulfate Soil

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ABSTRACT

Acid sulfate soil contains high acid cations. Palm oil mill effluent (POME) mixed with zeolite can increase pH of acid sulfate soil. Base cations (Ca, K and Mg) on zeolite are exchangeable with H^+ and Al^{3+} ions in acid sulfate soil. The aim of this study was to investigate the effects of application of POME-zeolite mixture on acid cations of acid sulfate soil. The study was arranged in a Completely Randomized Design, with 9 treatments of POME applied on 10 kg of acid sulfate soil. Acid sulfate soil was put into 45cm x 40cm polybag. POME plus zeolite powder (60 mesh, type clinoptilolite) was applied on soil by watering evenly and incubated for 4 weeks at room temperature. The results showed that the content of acid cations of exchangeable-Al and exchangeable-H decreased by 71.21% and 27.71%, respectively after application of POME -zeolite. The application of POME without zeolite decreased the content of exchangeable-Al and exchangeable-H by 46.54% and 42.75%, respectively. Aluminium saturation has decreased after POME-zeolite application from 42.68% (high) to 8.52% (very low). Base saturation increased after application of POME-zeolite application from 17.64% (very low) to 34.88% (low). Application of palm oil mill effluent-zeolite mixture decreased acid cations in acid sulfate soil. Base saturation correlates positively to pH and cation exchange capacity and negatively correlates to Al saturation. The main factors controlling the increase of pH, base saturation and cation exchange capacity of acid sulfate soil after POME-zeolite application were the decrease of exchange-able-H, followed by exchangeable-Al and total-Fe.

Keywords: Acid cations, acid sulfate soil, base cations, pH, POME, zeolite

ABSTRAK

Tanah sulfat masam memiliki tingkat kemasaman yang tinggi. Limbah Cair Pabrik Kelapa Sawit (LCPKS) dapat meningkatkan pH tanah dengan penambahan zeolit. Kation-kation basa Ca, K dan Mg pada zeolit dipertukarkan dengan ion H^+ dan Al^{3+} pada tanah sulfat masam. Pelaksanaan penelitian di Desa Ulu Lagan, Tanjung Jabung Timur, Provinsi Jambi, menggunakan Rancangan Acak Kelompok dengan 9 perlakuan dan 3 kelompok. Faktor perlakuan terdiri dari A = LCPKS+zeolit 10% (WPH 2 minggu) dosis 500 ml polybag^{-1}, B = LCPKS+zeolit 10% (WPH 2 minggu) dosis 750 ml polybag^{-1}, C=LCPKS +zeolit 10% (WPH 2 minggu) dosis 1000 ml polybag^{-1}, D = LCPKS+zeolit 5% (WPH 2 minggu) dosis 500 ml polybag^{-1}, E = LCPKS+zeolit 5% (WPH 2 minggu) dosis 750 ml polybag^{-1}, F= LCPKS+zeolit 5% (WPH 2 minggu) dosis 1000 ml polybag^{-1}. G= LCPKS (WPH 4 minggu) dosis 500 ml, H= LCPKS (WPH 4 minggu) dosis 750 ml, I= LCPKS(WPH 4 minggu) dosis 1000 ml. Zeolit dan LCPKS mempengaruhi pH tanah dan pH tanah berkorelasi positif sangat nyata dengan KTK dan C-organik serta berkorelasi negatif dengan Al-dd dan Fe total. Kejenuhan basa berkorelasi positif dengan pH dan KTK serta berkorelasi negatif dengan kejenuhan Al, korelasi negatif terjadi antara kejenuhan Al dengan pH, dan KTK . Faktor utama peningkatan pH, kejenuhan basa dan KTK tanah sulfat masam potensial setelah perlakuan LCPKS+zeolit adalah penurunan H-dd kemudian diikuti Al-dd dan Fe.

Kata Kunci: Kation asam, kation basa, pH, tanah sulfat masam, LCPKS, zeolit

INTRODUCTION

Palm oil mill effluent (POME) contains N, P, K, Ca, Mg, and various types of microbes that benefit for plant nutrition and soil amendment. Palm oil mill effluent can be used as organic fertilizer with the addition of zeolite. Application of 10% zeolite and hydrolysis detection time (HDT) within 2 weeks on POME acidic pool and application of 5% zeolite and HDT within 2 weeks on POME secondary anaerobic pool can produce better levels of N, P, K, Fe, Al, BOD and pH according to waste standard quality. The addition of zeolite followed by HDT treatment on POME will result in better N, P, K, Al, Fe, BOD and pH levels compared to zeolite treatment without HDT and HDT without zeolite treatment (Nursanti *et al.* 2013a).

Zeolite is negatively charged minerals, which can be neutralized by alkali metals, having pores filled with K, Na, Ca, Mg and H_2O molecules, allowing for the exchange of ions and the release of water back and forth. Besides being a cation exchanger, zeolite also serves as cation absorbent, such as Pb, Al, Fe, Mn, Zn, and Cu, so the zeolite can reduce heavy metal pollution in the environment (Oste *et al*. 2002; Wingenfelder *et al*. 2005).

Utilization of potential acid sulfate soils for agriculture is confronted with less supportive soil chemical properties due to low pH, high levels of Al, Fe, Mn and $SO4^{2-}$, high salt content, and deficiency of P, Cu, Zn, and B. These problems arise due to the presence of pyrite layer. This pyrite layer should be in the lower layers because if it is oxidized it can cause soil acidity and high levels of nutrient toxicity to plants (Fahmi and Hanudin 2008).

The increase of pH of acid sulfate soil is affected by zeolite. The basic cations present in zeolite such as Ca, K and Mg are interchangeable with H^+ and Al^{3+} ions. Zeolite can support soil pH, neutralize acid soils, adsorb Al and Fe causing soil acidity and release basic cations such as Ca, Mg and K (Endro 2008). Ca^{2+}, Mg^{2+}, K^+ cations in soil will be hydrolysed and produced hydroxide compounds and react with soluble Al ions in the soil solution to produce insoluble $Al(OH)_3$ (Ano and Ubachi 2007).

The application of 1000 mL POME plus 10% zeolite can increase the pH of acid sulfate soil from 4.10 to 7.03. The application of 500 mL POME plus 5% zeolite resulted soil pH of 4.27. The dose of 1000 mL POME plus 10% zeolite can increase soil organic-C from 1.76% to 3.12%. The soil P_2O_5 content increased from the low criteria (14.30 mg kg^{-1}) to high criteria (36.11 mg kg^{-1}) after application of 1000 mL POME plus 10% zeolite. Base saturation increased from 22.06% to 34.88% after application of 750 mL POME plus 10% zeolite. The content of exchangeable-Al and -H acid cations decreased about 43.69% and 16.67%, respectively after application of 1000 mL POME plus 10% zeolite, compared to that after treatment with 500 mL POME plus 5% zeolite. Aluminum saturation decreased from 22.16% to 8.52% after treatment with 1000 mL POME plus 10% zeolite compared to that after treatment with 500 mL POME plus 5% zeolite (Nursanti *et al*. 2013b).

The objectives of this study was to determine the correlation between soil pH and cation exchange capacity, base saturation, Al, Fe and organic-C, and to find out the main factors that play roles in increasing pH, cation exchange capacity, and base saturation of acid sulfate soil treated with POME-zeolite mixture.

MATERIALS AND METHODS

Research Design

Acid sulfate soil in Lagan Ulu Village, Geragai District, Jabung Timur Regency, Jambi Province is located at 1°11’58.66" N and 103°44’6.19" E. The study was conducted in 3 months. The study was arranged in a Completely Randomized Design with 9 treatments, namely A = 500 mL POME + 10% zeolite; B = 750 mL POME + 10% zeolite; C = 1000 mL POME + 10% zeolite; D = 500 mL POME + 5% zeolite; E = 750 mL POME + 5% zeolite; F = 1000 mL POME + 5% zeolite; G = 500 mL POME; H = 750 mL POME; and I = 1000 mL POME. Acid sulfate soil was taken at a depth of 0 - 30 cm and put into 45 cm × 40 cm polybag. Application of POME plus zeolite powder (60 mesh, type clinoptilolite) on 10 kg of acid sulfate soil was conducted by watering evenly and the soil was incubated for 4 weeks at room temperature.

Soil Analysis

Soil chemical analysis including available-P (Bray-1), total-Fe (diethylene triamine penta acetic acid/DTPA extraction), total-N (Kjedahl), exchangeable-K (NH_4OAc pH 7), exchangeable-Ca (NH_4OAc pH 7), exchangeable-Mg (NH4OAc pH 7), organic-C (Walkey and Black), CEC (NH_4OAc pH 7), S content (spectrophotometry), pH H_2O (1:1) and pH KCl (1:1) (electrometric method), exhangeable-Al (KCl extraction), and exhangeable-H (KCl extraction) was conducted.

Soil Characteristics

Soil layer observation at 20, 30, 40, 50, and 60 cm depths showed that the layers of acid sulfate soil are massive, unstructured, pale gray to dark gray, with groundwater level at 100 cm. Pyrite was observed at 30, 40, 50 and 60 cm depths with the contents of 0.08, 0.12, 0.13, 0.15 and 0.21%, respectively. The results of soil analysis (Table 1) showed that the soil was classified as clay texture, low cation exchange capacity, very acidic, low available-P and low total-N content.

Table 1. Characteristics of acid sulfate soil before treatment.

Parameter	value	Criteria *)
pH H_2O (1:1)	4.10	very acidic
pH KCl (1:1)	3.40	
EC (mS cm^{-1})	0.18	no salt
Organic C (%)	1.76	low
Total N (%)	0.18	low
C/N ratio	9.78	low
Available-P (mg kg^{-1})	14.30	low
K-total (mg kg^{-1})	43	high
Exc-Ca ($cmol_{(+)}kg^{-1}$)	1.08	very low
Exc-Mg ($cmol_{(+)}kg^{-1}$)	1.30	moderate
Exc-Na ($cmol_{(+)}kg^{-1}$)	0.98	high
Exc-K ($cmol_{(+)}kg^{-1}$)	0.47	moderate
Exc-H ($cmol_{(+)}kg^{-1}$)	1.38	low
CEC ($cmol_{(+)}kg^{-1}$)	15.24	low
Al-exc ($cmol_{(+)}kg^{-1}$)	4.34	
Al Saturation (%)	45.45	high
Base Saturation (%)	25.13	low
Fe (%)	1.61	
S (%)	0.15	

Note: *) based on the criteria proposed by Pusat Penelitian Tanah (1995).

Palm Oil Mill Effluent Characteristics

The POME used was secondary anerobic pool POME with pH of 6.82, total-N content of 0.18%, total-P of 0.07% and BOD of 3.01 g L^{-1}.

Data Analysis

The data were analyzed using the Analysis of Variance using Statistical Analysis System Version 17. The Duncan Multiple Range Test (DMRT) at 5% significance level was performed to compare the mean of each treatment. Furthermore, the correlation test was performed to determine the level of relationship between independent and dependent variables.

RESULTS AND DISCUSSION

The results of correlation analysis of several soil chemical properties after POME-zeolite application (Table 2) showed that the soil pH is positively correlated to CEC and organic-C and negatively correlated to exchangeable-Al and -Fe levels. The increase of pH will be followed by the increase of CEC and organic-C and the decrease of exchangeable-Al and Fe levels. The increase of CEC will be followed by the decrease of exchangeable-Al and Fe levels.

The application of POME-zeolite resulted in a higher base cation content in acid sulfate soil compared to that POME without zeolite application (Table 3). The highest content of base cations was measured in the treatment of 1000 mL POME + 10% zeolite with the level of moderate to very high, while the lowest content of base cations was measured in 500 mL POME without zeolite with very low to moderate level of base cations. The soil base saturation increased after POME+zeolite application (34.88%, low) compared to that in POME (without zeolite) application (17.64%, very low).

Table 2. Correlations of organic-C, CEC, pH, exchangeable-Al and -Fe due to the application of POME-zeolite on acid sulfate soil.

Variable	Correlation Coefficient Value			
	Org-C	CEC	Fe	Al
pH	+0.80**	+0.70**	-0.70**	-0.73**
Org-C	-	+0.54**	-0.67**	-0.68**
CE	-	-	-0.74**	-0.61**

Note: * *: very significant at 1% significance level.

Table 3. The amounts of exchangeable bases, base saturation (BS) and Al saturation of acid sulfate soil due to the application of POME-zeolite and POME without zeolite.

POME (mL) and Zeolite (%)	Exchangeable Base Cations				Exc -Al	Exc- H	BS	Al Saturation
	Ca	Mg	K	Na				
	---------------------cmol(+)kg^{-1}---------------------------						-----------%--------	
500 -10	3.87 l	2.61 h	1.41 vh	1.04 vh	1.82	0.66	28.49 l	15.94 l
750 -10	4.98 l	2.67 h	1.57 vh	2.04 vh	1.52	0.61	34.88 l	11.36 l
1000 - 10	5.49 m	2.74 h	1.66 vh	1.99 vh	1.16	0.60	34.85 l	8.52 vl
500 - 5	2.77 l	2.43 h	0.78 h	0.53 m	2.06	0.72	22.06 l	22.16 m
750 - 5	2.97 l	2.45 h	0.85 h	0.72 h	2.01	0.69	26.99 l	20.76 m
1000 -5	3.08 l	2.47 h	0.92 h	1.02 vh	1.92	0.68	25.07 l	19.01 l
500 - 0	1.66 vl	2.22 h	0.42 m	0.30 l	4.03	0.81	17.64 vl	42.68 h
750 - 0	1.82 vl	2.39 h	0.62 h	0.35 l	2.69	0.83	19.39 vl	30.99 m

Note: The letters in each column indicate the criteria for assessing the nature of the soil (LPT, 1983): vl = very low, l = low, m = moderate, h =high, vh = very high.

The results of correlation analysis indicated that the increase of organic-C is followed by the increase of pH and the decrease of exchangeable-Al. POME is a colloidal suspension containing dissolved organic materials and high solids thereby when applied to soil with low organic matter content, the organic matter content of the soil will increase with increasing dose of applied POME (Rashid *et al.* 2009).

Correlation analysis showed that the increase of organic-C is followed by the decrease of exchangeable-Al and -Fe (Table 2). The decrease of exchangeable-Al and -Fe is due to the role of dissolved organic materials in POME in the form of amino acids that can bind A1 and Fe cations to form insoluble Al-organic complex. Baharuexcin *et al.* (2009) showed that organic acids inhibit the liberation of A1 and Fe from the soil colloidal surfaces through the formation of stable Al-organic and Fe-organic complexes. The hydrolysis of Al and Fe ions can increase the concentration of H^+ ions in the soil to decrease soil pH. The formation of stable Al-organic and Fe-organic complexes by dissolved organic acids present in POME causes the decrease of A1 and Fe ion activity, thus preventing the occurrence of hydrolysis of Al and Fe ions.

The content of exchangeable-Al and -H cations decreased 71.21% and 27.71%, respectively after application of POME plus zeolite compared to that in the treatment of POME without zeolite. The application of non-zeolite POME can decrease the content of exchangeable-Al and -H cations by 46.54% and 42.75%, respectively compared to the initial Al and H cation levels prior to the experiment. Aluminum saturation decreased to 8.52% (very low) after POME+zeolite treatment compared to that in POME without zeolite, *i.e.* 42.68% (high) (Table 3).

Multiple correlation analysis of CEC, Base Satutarion (BS), pH and Al saturation due to the effect of POME-zeolite application (Table 4) showed that the base saturation is positively correlated to pH and CEC and negatively correlated to Al saturation. Negative correlations between Al saturation and pH, CEC and base saturation were observed. The increase of base saturation will be followed by the increase of pH and CEC and followed by the decrease of Al saturation. The increase of Al saturation will be followed by the decrease of pH, CEC and base saturation.

An increase of pH due to zeolite application is possible because the base cations present in zeolite such as Ca, K and Mg can be exchanged with H^+ and Al^{3+} ions. Zeolite can improve soil pH, acid soils can be neutralized because zeolite is neutral (pH 7.2) and can adsorb Al and Fe causing soil acidity and release base cations such as Ca, Mg and K. Kismolo *et al.* (2008) indicated that zeolite is a mineral that can neutralize soil pH.

The level of relationship of exchangeable-Al, exchangeable-H, and total-Fe with pH due to the application of POME-zeolite showed that the increase of acid sulfate soil pH due to the effect of exchangeable-H was greater than that of exchangeable-Al. The variance of exchangeable-H is 58.01%, exchangeable-Al is 33.41% and Fe is 8.57%. Thus the main factor controlling the increase of pH of acid sulfate soil is exchangeable-H then followed by exchangeable-Al and Fe. The lower the exchangeable-Al, exchangeable-H and Fe, the higher the soil pH is. The regression equation is presented below:

Table 4. Correlations of CEC, Base Satutarion (BS), pH, and Al saturation due to the application of POME-zeolite on acid sulfate soil.

Variable	Correlation Coefficient Value		
	pH	CEC	BS
BS	+0.79**	+0.49**	-
Al Saturation	-0.78**	-0.68**	-0.86**

Notes: * *: very significant at 1% significance level.

$$Y = -0.319x_1 + 0.927x_2 - 10.38x_3 + 12.423$$
$R^2 = 0.97$, Y = pH, x_1 = exc-Al, x_2 = total-Fe, x_3 = exc-H

Notohadikusumo and Noor (2003) described that agronomic constraints for agricultural crops cultivated on acid sulfate soils, among others, is the direct effect of acidity due to the increase of solubility and toxicity of Al, Mn and H^+; and phosphate deficiency due to Al-P precipitation. Muzar (2008) showed that POME application can increase pH of acid soil from pH 4.53 to 5.56.

The association of exchangeable-Al, exchangeable-H, and total-Fe with pH due to the application of POME without zeolite showed that the increase of acid sulfate soil pH due to the effect of exchangeable-Al was greater than that of exchangeable-H and total-Fe. The variance of exchangeable-Al is 53.84%, exchangeable-H is 40.96% and total-Fe is 5.20%. The regression equation is as follows:

$$Y = -12.672x_1 + 2.540x_2 - 0.251x_3 + 12.00$$
$R^2 = 0.94$, Y = pH, x_1 = exc-Al, x_2 = total-Fe, x_3 = exc-H

The increase of nutrient contents, such as total-N, available-P, and exchangeable base cations (Ca, Mg, and Na) are derived from mineralization of POME that releases these elements into the soil. In addition, the nutrient content is derived from POME decomposition and elements adsorbed by zeolite. Vaulina (2002) indicated that if the N content in the soil solution is less, then the N adsorbed on the zeolite will be released slowly. Palm oil mill effluent contains dissolved organic compounds that may play a role in increasing solubility of P present in acidic soils such as A1-P and Fe-P. The results of Ermadani *et al*. (2007) showed that application of organic acids (humic acids, oxalate and citrate) could increase available-P and total-P in soil.

Correlation analysis showed that an increase of organic-C content, total-N, available-P and base saturation will be followed by an increase of CEC and a decrease of Al saturation (Table 2 and 4). Wolf and Snyder (2003) indicated that the application of organic matter can increase the cation exchange capacity, soil pH and contribute a number of macro and micro nutrients through the process of mineralization. It is also explained that the function of organic matter in improving soil chemical fertility is also due to the decrease of nutrient losses by leaching due to the ion-binding by organic matter and immobilization of N, P and S and solubility of nutrients, especially phosphates and minerals by organic acids. The soil applied with POME-zeolite has a larger CEC compared to that applied with POME without zeolite, although both CEC are in the same criteria (Table 3).

This result is possible because the zeolite has a high CEC which is about 154.17 cmol(+) kg^{-1}. Li *et al*. (2003) showed that the exchange of cations in zeolite is essentially a function of the degree of silica substitution by aluminum in the structure of zeolite crystals. The more the amount of aluminum replaces the silica position, the more the negative charge is generated, so the CEC of zeolite will be higher. Acid sulfate soils have variable charges, if the pH rises above neutral it will produce a negative charge. Negative charges are generated by the high incidence of OH^- ions in the colloidal surfaces. Negative charges on the colloidal surfaces are able to adsorb the cations. Example of reaction is as follows:

$$Al\text{-}OH + OH^- \rightarrow Al\text{-}O^- + H_2O$$

The level of relationship of exchangeable-Al, exchangeable-H, and total-Fe with CEC due to the application of POME-zeolite showed that the increase of CEC of acid sulfate soil due to the effect of exchangeable-H was greater than that of exchangeable-Al and total-Fe. The regression equation is generated:

$$Y = 21.48x_1 - 2.027x_2 - 2.491x_3 + 23.176$$
$R^2 = 0.92$, Y = pH, x_1 = exc-Al, x_2 = total-Fe, x_3 = exc-H

Zeolite is a catalyst material that has the ability to improve the decomposition process of large molecular weight organic compounds (polymers) into simple organic compounds (monomers), which is supported by the nature of the zeolite electrostatic field and the role of pore space structure in zeolite. In addition, zeolite is the absorbing agent and pH neutralizer, which easily performs ion exchange (Ersoy and Celik 2003).

The adsorption process of ions that can cause soil acidity (Al^{3+}, Fe^{3+} and H^{+}) by zeolite occurs because there is an attraction between the ions that have a low electronegativity difference (Van Der Waals Style) and supported by the presence of cavities inside the zeolite which acts as a trap.

Level of relationship between exchangeable-Al, exchangeable-H and total-Fe with CEC due to the application of POME without zeolite shows that the decrease of CEC of acid sulfate soil due to the effect of exchangeable-H was greater than that of exchangeable-Al and total-Fe. The regression equation is presented as follows:

$$Y = -0.156x_1 + 1.309x_2 - 18.468x_3 + 39.717$$
R^2=0.82, Y= pH, x_1= exc-Al, x_2=total-Fe, x_3= exc-H

POME and zeolite are negatively charged. Application of POME and zeolite to soil means donating the negative charges to the soil, the higher the negative charge the higher the CEC will be, because the increase of the number of cations that are exchangeable. Furthermore, Fungaro and Graciano (2007) indicated that zeolite have open skeletons formed from primary builder units that further form secondary builder units. The morphology and structure of zeolite crystals consist of cavities that are connected in all directions that cause the surface of the zeolite to expand. There was a negative correlation between Al saturation with pH, CEC and base saturation. The increase in base saturation will be followed by the increase in pH and CEC and followed by the decrease in Al saturation. The increase of Al saturation will be followed by the decrease of pH, CEC and base saturation.

The increase of exchangeable base cations (Ca, Mg, and Na) is derived from the mineralization of POME that releases these elements into the soil. In addition, the nutrients are derived from POME decomposition and elements adsorbed by zeolite. Wolf and Snyder (2003) and Budianta *et al.* (2020) indicated that the application of organic matter can increase the cation exchange capacity, soil pH and contribute a number of macro and micro nutrients through the process of mineralization.

The level of relationship between exchangeable-Al, exchangeable-H and total-Fe with base saturation due to the application of POME-zeolite showed that the increase of base saturation of acid sulfate soil due to the effect of exchangeable-H was greater than that of exchangeable-Al and total-Fe. The regression equation is as follows:

$$Y = -3.150x_1 + 13.048x_2 - 80.67x_3 + 80.354$$
R^2 = 0.89, Y= pH, x_1 = exc-Al, x_2 = total-Fe, x_3 = exc-H

The level of relationship between exchangeable-Al, exchangeable-H, and total-Fe with base saturation due to the application of POME without zeolite showed that the increase of base saturation of acid sulfate soil due to the effect of exchangeable-H was greater than that of exchangeable-Al and total-Fe. The regression equation is as follows:

$$Y = -0.766x_1 + 1.237x_2 - 1.382x_3 + 23.22$$
R^2 = 0.97, Y = pH, x_1 = Al-exc, x_2 = Fe, x_3 = H-exc

CONCLUSIONS

The pH of acid sulfate soil applied with POME plus zeolite will increase, followed by the increase of CEC and organic-C content and the decrease of exchangeable-Al and total-Fe levels. The soil base saturation is positively correlated with pH and CEC and negatively correlated with Al saturation. There were negative correlations between Al saturation and pH, CEC and base saturation. The pH of acid sulfate soil increases due to the effect of decreasing exchangeable-Al and total-Fe levels. The lower the exchangeable-Al and total-Fe in the soil, the soil pH will increase. The main factors influencing the increase of pH, CEC and base saturation of acid sulfate soil applied with POME plus zeolite were exchangeable-H, followed by exchangeable-Al and total-Fe.

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